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(54) **Quenched nylon multilayer
casings**

(57) An exact-fit tubular food casing for pressurized filling comprises a tubular polymeric laminate having an outer layer of crystallized and moisturized nylon and an interior moisture barrier layer. In one advantageous mode, the nylon is heat crystallized from a substantially amorphous state and simultaneously moisturized. Associated method and apparatus for making said casing are also provided. In use, the casing is stuffed to within its elastic limit so that radial deformation is uniform along the length of the tubular casing. As the exposed nylon dries out, the casing shrinks, thus further elastically stretching tightly over the stuffed food roll contained therein.

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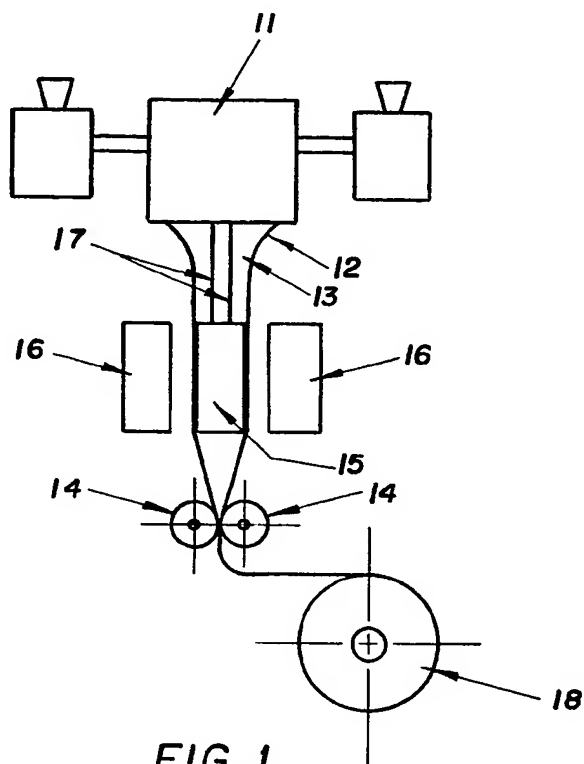


FIG. 1

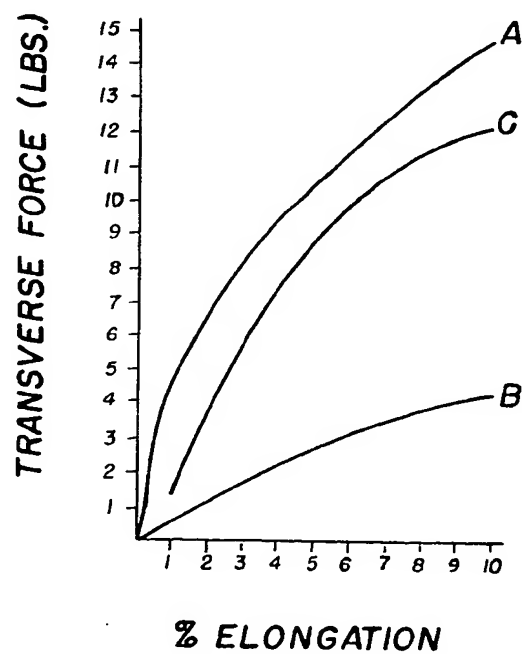


FIG. 2

SPECIFICATION

Quenched nylon multilayer casings

5 This invention relates generally to tubular food casings of laminated film and more particularly to casings that tightly encase a food product extruded or stuffed into the casing. 5

Many processed foods are stuffed into a casing during their processing, i.e. the foodstuff while in a fluid condition is flowed under pressure into a tubular casing. The food product may be further processed while encased in the casing or it may be stored in the casing pending 10 further processing or sale. For example, cheese rolls are typically formed by pressure filling a tubular food casing with a hot cheese melt or a solid curd cheese. The encased cheese is then allowed to solidify or knit, and under ideal conditions, the casing will have been filled uniformly to form a cheese roll of uniform diameter. 10

Ideally, these tubular casings possess material properties such that they are said to form an 15 "exact fit". This means that the casing radially deforms uniformly during pressurized filling so that the food roll formed therein has a substantially uniform diameter along its length. Further, nonuniform radial deformation is prevented, which weakens the casing thereby promoting bursting and spilling of the food product. Additionally, the term "exact fit" implies that the ideal casing has dimensional memory such that as the stuffed food product cools or knits contraction 20 of the casing is sufficient to maintain the casing securely and uniformly stretched over the shrunken food roll contained therein. 20

Conventionally, such casings are typically fibrous casings built up from a fibrous web of nonwoven paper, possibly with various synthetic polymer impregnations or laminations to achieve composite properties required for a particular application, such as low transmission of 25 oxygen and water vapor, as representatively shown by U.S. Patent 4,026,985. In use, these cellulosic casings must be soaked in hot water shortly before filling to render the casing sufficiently flexible and elastic for uniform filling from a shirred or gathered configuration and to wash away preservatives. There are a number of disadvantages in using this type of casing in that hot water soaking is required, while in the event of a cancelled run, soaked casings cannot 30 be saved. Further, the soak water is a source of contamination, and, due to relatively high moisture permeability, storage times of the stuffed casings must be relatively short to prevent food dryout. Significantly, these loaded casings are prone to bursting in a brittle failure mode. 30

In contrast, the present invention provides a casing that is not prone to brittle failure but rather will undergo considerable uniform plastic deformation without spilling the food stuffing if 35 the casing is inadvertently loaded beyond the elastic limit, while providing enhanced apparent elasticity thereby promoting a tighter, more uniform fit. Additionally, the casings may be moisturized well in advance of stuffing, yet will appear dry in the moisturized condition; however, should the casings be permitted to dry out, they may be resoaked, and may be thus cycled repeatedly. 35

Accordingly, the casing of the invention includes a non-fibrous, polymeric tubular laminate 40 having an outer layer of crystallized and moisturized nylon, thereby maximizing the elastic range and the elastic limit of the casing while maintaining a plastic failure mode. In one mode of use, the casing is stored in a humid environment to prevent dryout of the nylon outer layer. In an alternative mode, where the casings are permitted to dry out during storage, the nylon layer is 45 remoisturized prior to stuffing. Eventually, the casing is stuffed and is thereby subjected to a stuffing load to less than about its elastic limit so that radial deformation is uniform along the length of the tubular casing. As the exposed outer nylon dries out, the multi-ply casing surprisingly shrinks uniformly, thus further elastically stretching tightly over the stuffed food roll contained therein. This contraction on dry out yields, in effect, an apparent increase in the 50 elasticity of the casing. In the case where the food stuffing is hot, this delayed shrinkage offsets thermal contraction on cooling of the food stuffing, thereby providing a wrinkle-free tight package. 50

In the method aspects of the invention, there is provided a method of making an exact-fit food casing, which comprises:

- 55 a) melt-forming a nascent tubular polymeric laminate having an outer nylon layer; 55
- b) quenching and solidifying said nascent tubular laminate, such that said nylon is rendered substantially amorphous;
- c) heat treating said tubular laminate to substantially crystallize said nylon; and
- d) moisturizing said nylon.

60 In one advantageous mode, the method comprises melt-forming a nascent tubular polymeric laminate having an outer nylon layer; then quenching and solidifying the nascent tubular laminate, such that said nylon is rendered substantially amorphous, while simultaneously sizing the tubular diameter to about equal to a predetermined end-use diameter; and then heat treating said tubular laminate to substantially crystallize said nylon, while simultaneously moisturizing to 65 substantially saturate said nylon. 65

The invention also provides an exact-fit casing, comprising a tubular melt-formed polymeric laminate having an outer layer of crystallized and moisturized nylon and an interior moisture barrier layer, said nylon layer having been heat crystallized from a substantially amorphous state and simultaneously moisturized. In one advantageous embodiment, the casing is further

5 characterized in that said nylon has been crystallized from a substantially amorphous state and simultaneously water saturated, further provided that said nylon was rendered substantially amorphous while sizing said tubular laminate to a predetermined end-use diameter, the thickness of said nylon having been selected so that the elastic range of the force-elongation profile of said casing is at least about coextensive with the range of end-use loading. 5

10 In the apparatus aspects of the invention, there is provided apparatus for making an exact-fit food casing, comprising: 10

a) means for melt-forming a nascent tubular polymeric laminate having an outer nylon layer and an interior moisture barrier layer;

b) means for quenching and solidifying said nascent tubular laminate, such that said nylon is rendered substantially amorphous, while simultaneously sizing the tubular diameter to about equal a predetermined end-use diameter; and 15

c) means for heat treating said tubular laminate to substantially crystallize said nylon while simultaneously moisturizing to substantially saturate said nylon. In one advantageous mode, said apparatus further includes means for quenching said nascent tubular laminate while simultaneously sizing said laminate to a diameter about equal to a predetermined end-use diameter; and 20 means for simultaneously heat treating and moisturizing said nylon. 20

Further details are given below with reference to the drawings wherein:

Figure 1 is a schematic flowchart of a preferred mode of making the casing of the invention; and

25 *Figure 2* is a stress-strain diagram comparing a casing of the invention to a conventional fibrous casing in the dry and soaked conditions. 25

The laminate structure of the present invention is directed to a multilayer tubular food casing having an outer nylon layer adhered over one or more inner layers including one or more moisture barrier layers, for example the structure nylon(outer)/adhesive/barrier(inner). Conventionally, nylon is present in food casing laminates to serve as an oxygen barrier to impede the inward diffusion of oxygen and to impart relatively high strength to the laminate, while a polyolefin, such as polyethylene, EVA, saran, or surlyn, including copolymers and terpolymers thereof is used as an inner surface being relatively moisture impermeable and chemically inert to many food stuffs. It is emphasized, however, that the foregoing composite arrangement is

30 exemplary and that the casing of the invention comprehends multiple interior layers as may be selected to achieve the desired composite properties as required in a given application, subject to the essential features of the invention. As discussed below, it is essential that the nylon layer be formed as the outer layer of the casing to render the nylon immediately susceptible to selected heat treatment, moisturizing, and drying conditions. According to an especially 35 advantageous mode of the invention, the nylon is further characterized according to selected crystalline microstructure as developed by selected process steps. This selected microstructure is manifested by a selected stress-strain profile, as further discussed below, exhibiting enhanced yield point and elastic elongation, and moisture absorptivity, relative to prior art tubular food casings, while maintaining in the moisturized condition a plastic, nonbrittle failure mode with 40 substantial plastic elongation. Of general interest, on the subject of developing selected stress-strain profiles in tubular food casings, are the casings of U.S. Patent 4,303,711 wherein it is disclosed that the elastic range of a casing is extended to near the ultimate stress by selected processing steps involving biaxial orientation, thereby diminishing the susceptibility of the casing to irreversible plastic deformation during pressurized filling of the casing. 45

Advantageously, the nylon layer of the casing of the invention is composed of a heat crystallizable nylon having a moisture absorptivity of at least about 8%, such as nylon 4, 6, or 66, and more preferably about 15%. A preferred laminate structure for the casing is nylon 6 or 66/Plexar/polyethylene. The adhesive Plexar (TM) is commercially available from the Chemplex Company. Plexar variants are described in U.S. Patents 4,087,587, 4,087,588 and 4,303,711 cited above. Plexar 2 adhesive may be generally characterized as an adhesive of the type comprising blends of a graft copolymer of a high density polyethylene and at least one unsaturated, fused ring, carboxylic acid anhydride and this blended with one or more resin copolymers of ethylene and an ethylenically unsaturated ester. Plexar 3 is preferred in the foregoing embodiment, comprising blends of a graft copolymer of a high density polyethylene and at least one unsaturated fused ring carboxylic acid anhydride blended with a polyethylene resin of one or more homopolymers of ethylene, copolymers of ethylene and an alpha-olefin or any or all of these. More generally, adhesives are suitable in the invention that comprise a chemically modified polyolefin selected from the group consisting of ethylene vinyl acetate polymer, high density polyethylene and rubber modified high density polyethylene, each 50

55 chemically modified by the provision of functional groups to the polymer which have a strong 55

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65 65

affinity for nylon and which will form a strong bond to nylon under heat and pressure of coextrusion as representatively shown in U.S. Patent 4,233,367. An alternative structure, though less preferred, utilizes in part the technology described in U.S. Patent 4,104,404 for "Cross-Linked Amide/Olefin Polymeric Tubular Film Coextruded Laminates", wherein amide/olefin films are disclosed that are relatively resistant to delamination when used in heated conditions. The adhesive utilized is of the type that has irradiatively cross-linkable monomeric units, the major component being olefin units, further provided that the olefins in the adhesive and in the polyolefin layer are the same.

Referring specifically to the drawings, in Fig. 1 a preferred method of making the casing of the invention is schematically illustrated. The multilayer casing is tubularly coextruded through a conventional die 11, for example at a rate of about 25 to 80 fpm (0.13 to 0.41 m./s) with the nascent tube 12 being lightly drawn from the die as indicated at 13. The nascent tube is lightly drawn by nip rollers 14 over a sizing mandrel 15 and through quench means 16 which provide quenching by water cascading from a conventional water ring, or by water flooding or spraying. The extrusion rate is sufficient such that the nascent tube enters the quench zone with the outer nylon layer remaining in the substantially amorphous condition. The quench means 16 are sufficient in capacity to quench the nylon layer to maintain the as extruded amorphous condition within the nylon to below its glass transition temperature. Preferably, the sizing mandrel 15 is internally cooled to augment the quench rate, as indicated by coolant lines 17. As further discussed below, it has been discovered that the diameter of mandrel 15 is selected approximately according to the desired end use diameter of the casing, i.e. according to the desired diameter of the food roll to be contained therein. After quenching, the advancing casing is collapsed and drawn through nip rollers 14 and passes in line to take up roll 18. Rolls of casing are then heat treated in the presence of moisture at a temperature and for a time sufficient to substantially crystallize the amorphous nylon layer of the casing. Preferably, heat treatment is conducted by submersion of the filled roll in a hot water bath (not shown). Representatively, such heat treatment is continued for about 1 to 12 hours, preferably about 1 to 4 hours, depending upon the extent of crystallization desired, and at a temperature in the range of about 180 to 212°F (82 to 100°C). Alternatively, heat treatment may be conducted by continuously passing the advancing tubing directly through a heat treatment medium before winding on the take up roll so that a shorter treatment time is suitable, representatively about 1 minute to 1 hour, owing to the relatively rapid response of the exposed nylon to heat treatment. The foregoing method is seen to be especially suited for unitary formation of the casing of the invention. In an alternate mode, though less preferred, the nylon layer may be melt-formed as a single layered sheet, crystallized and moisturized, laminated with the desired substrate, and then formed into a tube.

The term "crystalline" is used in the conventional sense to refer to the existence of long range three dimensional atomic order or periodicity on the atomic scale within a material. The term "amorphous" is also used in the conventional sense as synonymous with the term "non crystalline" to refer to the absence of long range atomic periodicity within a material. The degree of atomic order may be determined by conventional techniques, such as by x-ray diffraction techniques to determine radial atomic probability density. As a first approximation, a material may be characterized as substantially crystalline if the material possesses a definite melting temperature or range, whereas a material may be characterized as substantially amorphous if the material gradually becomes less viscous on heating without exhibiting a definite melting point or range.

The casings are ultimately used in forming a food roll or log wherein a paste-type food stuff, such as a hot cheese melt, is stuffed under pressure into a casing cut and clipped to the desired length. The casings are typically stuffed from a shirred configuration on conventional food stuffing apparatus, as representatively shown in U.S. Patent 4,307,489 directed to preparatory treatment of gathered casings. Thus, flexibility of the casing is seen to be important for uniform filling of the casing. In the present invention, the fresh casings following the above discussed heat treatment steps have been thoroughly moisturized and thus are in a relatively flexible condition. Preferably, the casings are packed, cut and clipped in a lay-flat stack and are stored in a substantially saturated humid environment to prevent dryout. Alternatively, if the casings are permitted to dry out during storage, they are remoisturized prior to stuffing to restore flexibility. During stuffing, the cheese melt is extruded into the shirred casing under a pressure such that the casing is stressed within its elastic range, thereby preventing nonuniform plastic deformation of the casing with resultant non-uniform stuffing and irregular formation of the cheese roll. Advantageously, the thickness of the nylon layer is selected so that the elastic range of the force-elongation profile of the casing is at least about coextensive with the range of end-use loading.

The stuffed casing is then clipped and permitted to cool and dry. Contraction of the food product on cooling or curing tends to loosen the fit of the casing on the food product; however, on dryout of the nylon layer of the casing, representatively occurring over about 0.5 hour, the

casing contracts, due to residual elasticity and dry out, thereby offsetting thermal contraction of the cheese roll to maintain a tight, wrinkle free fit. Thus, the casing may be said to have enhanced apparent elasticity over the elastic range as conventionally defined. As stated above, a key result of the invention involves the relationship between the diameter of mandrel 15 and the

5 finally loaded diameter of the casings, namely the loaded and dried casing diameter will be about equal to the selected diameter of mandrel 15. For example, casings were made by the above process utilizing a mandrel diameter of 3.243 inches (8.24 cm) and having a composite structure of 2 mils (0.05 mm) nylon 6/0.5 mil (0.013 mm) Plexar 3/1.5 (0.038) mils LDPE (inner). The fresh casings after heat treatment and remaining in the saturated condition had a

10 diameter of 3.104 inches (7.9 cm), and after drying had a diameter of 3.024 inches (7.7 cm). The moisturized casings were stuffed with a cheese melt at about 160°F (71°C) to a diameter of 3.275 inches (8.3 cm). The loaded casings after cooling and dryout had a diameter of 3.25 inches (8.25 cm). Thus, the finally loaded casing diameter was very nearly equal that of sizing mandrel 15. Further, it should be noted that the casing exhibited a dry out contraction of about

15 3%, a substantial amount relative to the elastic range of the casing. Additionally, the multiply casing was observed to undergo this contraction in a unitary manner, i.e. the overall casing followed the outer nylon layer without wrinkling or separation of the component layers.

Further, the casing of the invention as compared to conventional fibrous casings has been found to have substantial advantages in other respects. Water soaking immediately prior to

20 stuffing is eliminated thereby eliminating surface water on the casing. Even though the nylon may be in the saturated condition it will not be visibly wet, thereby eliminating wet conditions about the stuffing machine area. The casing has been found to have dimensional stability along its length, for example the loaded diameter was found to be within 0.02 inch (0.5 mm) tolerance thereby forming a substantially uniform cheese roll. In the event the elastic range of

25 the casing is exceeded during stuffing, the casing elongates plastically and does not fail brittly, thereby preventing food product breakout and waste. For example, the casings of the invention have been found to have a typical plastic elongation of about 500%, as compared to an elongation of about 20% for the above mentioned conventional casings. On storage, the food fill does not dry significantly since the casing of the invention is nonporous, thereby affording

30 considerable shelf life. The moisturized condition of the casings of the invention is reversible, so that the casings may be remoisturized prior to stuffing in the event that the casings dry out during storage. Since this process is reversible for the casings of the invention, waste of the casings is prevented in the event the food stuffing process is interrupted for a substantial period of time. Finally, due to the enhanced apparent elasticity of the casing affording a tight second

35 skin fit, the food product contained therein is left with an appealing semi-glossy surface after stripping the casing from the food roll.

In Fig. 2, there are shown transverse force-elongation profiles in the elastic range comparing the above described exemplary casing of the invention to a conventional non-woven fibrous casing. Profiles A and B show the elastic response of the conventional casing in the dry and wet

40 conditions, respectively. The conventional casings must be stuffed in the wet condition and thus are relatively susceptible to overloading and bursting since profile B is substantially less than A. Profile C refers to the elastic profile of a typical moisturized casing of the invention which is seen to be comparable to the substantially higher strength of the conventional casing in the dry condition.

45 There are several aspects of the invention that bear emphasis. As stated above, the casings of the invention have an enhanced apparent elastic range manifested as the moisturized external nylon layer of the stuffed casing dries out and shrinks, thereby promoting a tight, uniform fit. Another aspect involves enhanced yield strength and enhanced elastic range arising from the crystallizing heat treatment as discussed above. Each of these enhancements serves to extend

50 the elastic range of the casing thereby promoting dimensional stability during stuffing, i.e., a lessened susceptibility to non-uniform, plastic, irreversible deformation or bulging of the stuffed casing. Advantageously, this enhancement of the elastic range is not at the expense, however, of plastic elongation should the casings be inadvertently overloaded, in which event the casings will fail plastically and not brittly. It is believed that the favorable mechanical properties of the

55 casings of the invention may be further enhanced by the manner of crystallization of the outer nylon layer. First, in the preferred mode the nylon is crystallized on reheating from the substantially solid amorphous, cooled condition, as opposed to crystallization from the melt. At the least, a highly reproducible process for making the casings is provided in that the condition of the nylon just prior to heat treatment is definite. Second, crystallizing heat treatment is

60 preferably conducted in the presence of moisture which is believed to effect the mode of crystallization as well as desirably promoting high moisture absorptivity and plasticity of the nylon. More generally, the broad aspects of the invention involve crystallization of the outer nylon layer to enhance mechanical properties in the elastic range and moisturizing of the nylon layer to enhance elastic flexibility and plastic extensibility and to provide additional apparent

65 elasticity on dryout, with the surprising overall result that the multi-ply casing exhibits these

TABLE II: PROPERTIES AT 73°F (23°C)

Sample Type	YS-Transverse Yield Strength, psi, and (Standard Deviation) [kPa and standard deviation]				E-Transverse Elongation at Yield, %, and (Standard Deviation)			
	A		Condition B		C			
SDX244	YS	2770	(70)[19100/480]	2990	(30)[20600/210]	3130	(24)[21600/170]	
	E	14		>25		24	(1)	
SDX246	YS	2809	(90)[19300/620]	2470	(190)[17000/1300]	2180	(90)[15000/620]	
	E			24	(0)	25		

CLAIMS

1. A method of making an exact-fit food casing, which comprises:
 - a) melt-forming a nascent tubular polymeric laminate having an outer nylon layer;
 - b) quenching and solidifying said nascent tubular laminate, such that said nylon is rendered substantially amorphous;
 - c) heat treating said tubular laminate to substantially crystallize said nylon; and
 - d) moisturizing said nylon.
2. A method according to claim 1 wherein the said heat treatment and moisturization are conducted simultaneously and before substantial natural aging of the said nylon occurs.
3. A method according to claim 2 wherein the said nylon is substantially saturated by the moisturization.
4. A method according to claim 2 or 3 wherein the said heat treatment and moisturization are conducted in a hot water bath.
5. A method according to any of claims 1 to 4 wherein the said heat treatment is conducted at 180 to 212°F (82 to 100°C) for 1 minute to 12 hours.
6. A method according to any of claims 1 to 5 further comprising storing the said tubular laminate in a humid environment pending use.
7. A method according to any of claims 1 to 6 wherein the said quenching is conducted while the said tubular laminate is simultaneously sized to a diameter about equal to a predetermined end-use diameter.
8. A method according to claim 7 wherein the said nascent tubular laminate is quenched while passing over a mandrel of about said predetermined end-use diameter.
9. A method according to claim 8 wherein the said mandrel is internally cooled.
10. A method according to claim 8 or 9 wherein the said quenching is water quenching.
11. A method according to any of claims 1 to 10 wherein the said melt-forming is by coextrusion.
12. A method according to any of claims 1 to 11 wherein the said nylon comprises a heat crystallizable nylon having a moisture absorptivity of at least 8% and the said tubular laminate comprises an inner moisture barrier layer.
13. A method according to claim 12 wherein the composite structure of said tubular laminate comprises nylon (outer)/adhesive/polyolefin (inner).
14. A method according to claim 13 wherein the said nylon comprises nylon 6 or nylon 66.
15. A method according to claim 13 or 14 wherein the said polyolefin comprises polyethylene and the said adhesive comprises a chemically modified polyolefin selected from ethylene/vinyl acetate polymer, high density polyethylene, and rubber-modified high density polyethylene, each chemically modified by the provision of functional groups to the polymer which have a strong affinity for nylon and which will form a strong bond to nylon under heat and pressure of coextrusion.
16. A method according to claim 1 substantially as hereinbefore described with reference to Fig. 1 of the accompanying drawing.
17. An exact-fit food casing when produced by the method of any of claims 1 to 16.
18. An exact-fit food casing, comprising:
 - a tubular melt-formed polymeric laminate having an outer layer of crystallized and moisturized nylon and an interior moisture barrier layer, said nylon layer having been heat crystallized from a substantially amorphous state and simultaneously moisturized.
 19. A casing according to claim 18 wherein the thickness of the said nylon layer is selected so that the elastic range of the force-elongation profile of said casing is at least about coextensive with the range of end-use loading.

20. A casing according to claim 18 or 19 wherein the said nylon has been preliminarily rendered substantially amorphous while sizing said tubular laminate to a predetermined end-use diameter.
21. A casing according to any of claims 18 to 20 wherein the said nylon comprises a heat crystallizable nylon having a moisture absorptivity of at least 8%. 5
22. A casing according to claim 21 wherein the structure of said tubular laminate comprises nylon (outer)/adhesive/polyolefin (inner).
23. A casing according to claim 22 wherein the said nylon comprises nylon 6 or nylon 66.
24. A casing according to claim 22 or 23 wherein the said polyolefin comprises polyethylene and the said adhesive comprises a chemically modified polyolefin selected from ethylene/vinyl acetate polymer, high density polyethylene and rubber-modified high density polyethylene, each chemically modified by the provision of functional groups to the polymer which have a strong affinity for nylon and which will form a strong bond to nylon under heat and pressure of coextrusion. 10
25. Apparatus for making an exact-fit food casing, comprising: 15
- a) means for melt-forming a nascent tubular polymeric laminate having an outer nylon layer and an interior moisture barrier layer;
- b) means for quenching and solidifying said nascent tubular laminate, such that said nylon is rendered substantially amorphous, while simultaneously sizing the tubular diameter to about equal a predetermined end-use diameter; and 20
- c) means for heat treating said tubular laminate to substantially crystallize said nylon while simultaneously moisturizing to substantially saturate said nylon.
26. The apparatus of claim 25 wherein the said means for melt-forming comprise means for coextrusion.
27. The apparatus of claim 25 or 26 wherein the said means for sizing said laminate comprises a mandrel of about said predetermined end-use diameter. 25
28. The apparatus of claim 27 wherein the said mandrel is internally cooled.
29. The apparatus of any of claims 25 to 28 wherein the said means for quenching comprise means for water quenching.
30. The apparatus of claim 25 substantially as hereinbefore described. 30

